#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Ting WANG, Meng-Jiao WANG, Glendon A. McCONNELL & Steven R. REZNEK

SERIAS NO. 10/010,764

CONFIRMATION No. 3002

FILED: 7 DECEMBER 2001

For: "ELASTOMER COMPOSITES, ELASTOMER BLENDS AND METHODS"

The Commissioner of Patents Washington, D.C. 20231 Attn: Box Missing Parts

#### PETITION UNDER 37 CFR 1.47(a)

Dear Sir:

Petition is hereby made for filing the above-identified application when one of the joint inventors, namely: Glendon A. McConnell, refuses to sign and/or cannot be found.

Mr. McConnell was an employee of Cabot Corporation ("Cabot") at the time the invention was made. A copy of the confidentiality and proprietary rights letter agreement that Mr. McConnell signed with Cabot on or about the time he became a Cabot employee is attached hereto as Exhibit A. Mr. McConnell resigned from Cabot, effective 1 March 2002.

On 21 March 2002, Mr. McConnell sent an e-mail to Mr. Louis Grasso of Cabot's Human Resource Department to provide Cabot with his forwarding address:

3031 La Salle Avenue Apt. #4 Rockford, Illinois 61114 RECEIVED

JUL 1 8 2002

OFFICE OF PETITIONS

#### **CERTIFICATION UNDER 37 CFR 1.10**

I hereby certify that this paper (along with any document or paper referred to herein) is being deposited with the United States Postal Service on this date, 12 July 2002, in an envelope as "Express Mail Post Office to Addressee" Mailing Label Number EL 886 078 797 US, addressed to: Box Missing Parts, The Commissioner for Patents, Washington, D.C. 20231.

07/17/2002 AWONDAF1 00000042 030060 10010764

06 FC:122 130.00 CH

Name of person mailing paper

Ellen R. Ringland

Signature

Docket No. 01057 PATENT

(A copy of the e-mail is attached hereto as **Exhibit B**.)

On 30 May 2002, the undersigned attorney sent a letter to Mr. McConnell, at the above-stated address, by "Express Mail Post Office to Addressee," requesting signature of the *Declaration and Power of Attorney* and *Assignment* for the above-identified patent application and enclosing a copy of the above-identified patent application with the *Declaration and Power of Attorney* and *Assignment* therefor. The Express Mail package was delivered to on 31 May 2002 to Keith McConnell, who signed for the package. (A copy of the letter and enclosures, Express Mail mailing label, and signed delivery receipt are attached hereto as Exhibit C.)

When no reply was received after about two weeks, a follow-up letter was sent by the undersigned attorney to Mr. McConnell at the same address by FedEx on 13 June 2002. According to the FedEx tracking information, the FedEx package was delivered to a different address and signed for by D. Sengulb on 14 June 2002. (A copy of the letter, FedEx shipment details, and tracking report for delivery of the package is attached hereto as Exhibit D.)

When no reply was received after one additional week, an e-mail was sent to Mr. McConnell by the undersigned attorney on 21 June 2002 at the e-mail address shown on Exhibit B, to follow up on the earlier letters that were sent. (A copy of the e-mail is attached hereto as Exhibit E.)

In a call to directory assistance to obtain telephone information for a listing under "McConnell" on "LaSalle Avenue" in "Rockford, Illinois", the undersigned attorney received a recording that, at the customer's request, that number is nonpublished and not listed in their records.

To date, no reply has been received by the undersigned attorney to any of the letters or e-mail sent to Mr. McConnell.

It is respectfully requested that this Petition be granted and that the *Declaration and Power of Attorney* executed by Ting Wang, Meng-Jiao Wang, and Steven R. Reznek, and

Docket No. 01057 PATENT

unsigned by Glendon A. McConnell (and setting forth his last known address and citizenship) be accepted in satisfaction of the requirements of 37 C.F.R. 1.47(a).

Please charge the Petition fee of One Hundred Thirty Dollars (\$130.00) and any other fee that may be due in connection with this Petition to Deposit Account 03-0060. (A duplicate of this Petition (without attachments) is enclosed.)

Date: 12 July 2002

Respectfully submitted,

Martha Ann Finnegan (31,453) Attorney for Applicants

CABOT CORPORATION 157 Concord Road Billerica, Massachusetts 01821 Tel. No. (978) 670-6198 Fax No. (978) 670-8027

# **EXHIBIT A**

Glen McConnell	5/6/96
(Name) Glen mc Connell	(Date)
(Street Address)	(Hiring Location)
Gilling MA (State)	337-72-86/6 (Social Security No.)

Dear

Cabot Corporation, together with its subsidiaries and affiliates and any subsidiaries or affiliates which may be formed by any of them in the future (hereinafter collectively referred to as "Cabot"), is and expects in the future to be actively engaged in research and development, design, construction work and commercial operations using or relating to methods, processes, apparatus, and inventions. Cabot maintains as confidential and as trade secrets certain information, including computer programs, concerning both its research and development work and its commercial operations generally. During the period of your employment with Cabot you may have access to and acquire knowledge of such confidential information and trade secrets concerning the activities of Cabot. When executed and returned as provided below, this letter will constitute an agreement with respect to such matters and will supersede and replace any other prior agreement with Cabot relating to such matters.

In consideration of your employment by Cabot, you agree:

1. To maintain in confidence at all times during and after your employment any knowledge which you acquire in the course of your employment with Cabot or which you may acquire after such employment as to the confidential information, technical know-how, and trade secrets of Cabot, and also as to confidential records, drawings, data, methods, programs, processes,

Form 101R: 3872g-1

 Cabot Corporation

 950 Winter Street

 P. O. Box 9073

 Waltham, Massachusetts 02254-9073

 (617) 890-0200

apparatus, and inventions of Cabot. You further agree that you will not use any of said knowledge, information, know-how, and trade secrets independently of your work for Cabot and will not divulge the same to others. You further agree to surrender all papers, notes, drawings, transparencies, records, tapes, discs, models, and materials, and copies thereof, of every kind in your possession or control pertaining to said knowledge, information, know-how, or trade secrets upon termination of your employment with Cabot.

- 2. To assist Cabot in all possible ways in the discovery, perfection, and development of any methods, programs, processes, apparatus, and inventions (whether patentable or not) relating to the activities of Cabot or relating to any subject matter with which your work for Cabot is or may be concerned, all for the benefit of Cabot and as its exclusive property, compensation for all of which is hereby acknowledged to be included in the remuneration paid you by Cabot.
- 3. To disclose promptly all conceptions, improvements, and inventions made by you or disclosed to you by another Cabot employee during the period of your employment with Cabot to your immediate superior or to an executive officer of Cabot. You further agree that any conception, improvement, or invention relating to the actual or contemplated activities of Cabot or relating to any subject matter with which your work for Cabot was concerned, which, within twelve months after termination of your employment, you have disclosed to anyone or on which you have filed application for letters patent, shall be presumed to have been made by you either solely or jointly with others during your period of employment with Cabot.
- 4. To assign to Cabot, its successors, assigns, and/or designees, all of your rights to any and all improvements and inventions (whether patentable or not) which, during the period of your employment with Cabot or thereafter under the presumption of preceding paragraph 3, you make or conceive, either solely or jointly with others, relating to the actual or contemplated activities of Cabot, including all rights of priority under the International Convention for the Protection of Industrial Property and related conventions. You further agree, without charge to Cabot but at its expense, to execute, acknowledge, and deliver all such further documents, including applications for patents and assignments, as may be necessary or desirable to obtain patents in any and all countries and to vest full and complete title thereto in Cabot, its successors, assigns, and/or designees and to assist them at their expense in the enforcement of such patents.

Form 101R: 3872g-2.

- 5. That, should you elect to terminate your employment with Cabot, you will use your best efforts to give Cabot at least 30 days' advance written notice of such termination and will identify your subsequent employer, if any, and the nature of the work in which you expect to be engaged therewith, in such notice. In any event, whether termination of your employment is voluntary or involuntary, you hereby give permission to Cabot to communicate with your new employer for the purpose of advising your new employer of those secrecy obligations which you continue to have to Cabot.
- That you will not, for a period of one year from the date of termination of your employment with Cabot, become associated with or employed by any individual, firm, or corporation in any capacity involving research in, production of, design or engineering of or sales of any product or element thereof, or any processes or facilities for the production of any product or element thereof, (i) which is or may become competitive with any product which is the subject of Cabot's research, design, development, manufacture, or sale prior to the time of your termination, and (ii) with respect to which product or element thereof, or processes or facilities for the production thereof, you possess or have had access to confidential information, technical know-how, or trade secrets of Cabot. You further agree that if Cabot so requests, you will not become associated with or employed in any capacity by any individual, firm, or corporation engaged or planning to engage in activities competitive with those activities of Cabot with respect to which you possess or have had access to confidential information, technical know-how or trade secrets of Cabot for such period, not in excess of one year, as Cabot shall specify, provided that, during that period Cabot shall continue to pay you compensation at the rate of one-half of the base salary or wage being paid to you at the time of your termination.
- 7. That you will honor any presently effective secrecy agreement with former employers (copies of which agreement, if available, you have attached hereto). Cabot assures you that you are not and will never be required as a condition of employment to disclose any confidential information, technical know-how, or trade secrets of a proprietary nature of any former employer and you agree not to disclose any such information, know-how, or trade secrets to Cabot.
- 8. That determination that any provision of this agreement is invalid or unenforceable shall in no manner affect the validity or enforceability of any other provision of this agreement.

Form 101R: 3872g-3

This letter will constitute an agreement between us upon your signing three copies of it in the space provided below and returning two copies to Cabot. The third copy is for your files.

Very truly yours,

CABOT CORPORATION

Samuel W. Bodman

Chairman of the Board

I hereby agree to the terms set forth above:

Cimpture.

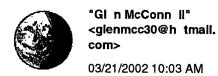
Glen McConnell

Date:

5/6/9/6

Form 101R: 3872g-4

# **EXHIBIT B**



To: <louis\_grasso@cabot-corp.com>

CC

Subject: G. McConnell: Forwarding Address for W-2 Form

Bud,

I just realized that Cabot does not have a forwarding address on file for sending my 2002 W-2 form to me at the end of the year. Could you please arrange for Cabot to send my W-2 form to the following address:

Glen McConnell 3031 La Salle Ave. Apt. #4 Rockford, IL 61114

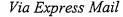
Thank you, in advance, for taking care of this detail.

Regards,

Glen

Get more from the Web. FREE MSN Explorer download: http://explorer.msn.com

# **EXHIBIT C**





30 May 2002

# CABOT CONFIDENTIAL

Martha Ann Finnegan
Chief Intellectual Property Counsel

Glendon A. McConnell 3031 LaSalle Avenue Apt. #4 Rockford, Illinois 61114

RE: U. S. Patent Application Serial No. 10/010,764

Filed: 7 December 2001

Entitled "Elastomer Composites, Elastomer Blends And Methods"

Our Reference No: 01057

## Reply Requested As Soon As Possible

#### Dear Glen:

Enclosed are copies of the above-referenced U.S. Patent Application Serial No. 10/010,764, which was filed in the U.S. Patent & Trademark Office on 7 December 2001, together with an original *Declaration & Power of Attorney* and *Assignment* therefor. This application is based upon Cabot RID No. CBK01057.

Please carefully read the copy of the patent application and carefully read the *Declaration & Power of Attorney* relating thereto.

When you sign the Declaration & Power of Attorney, you are declaring that:

- (a) you believe that you are an inventor of the subject matter which is claimed in the referenced application;
- (b) you have reviewed and understand the contents of the referenced application, including the claims, as amended by any amendment that is specifically referred to in the Declaration and Power of Attorney; and
- you will comply with the duty to disclose information to the Patent Office which is material to patentability (as defined in 37 CFR 1.56).

Glendon A. McConnell 30 May 2002 Page 2

Information is material if it establishes, by itself or together with other information, that any claim is not patentable. Information is also material if it refutes or is inconsistent with any statement made in the application that was provided to support or illustrate the patentability of any claim. Information can be in the form of publications, patents, and products that are/were available for sale, other public information, etc.

You can satisfy your duty by furnishing me with any such information. If you are aware of any such information please provide it to me as soon as possible. This duty continues as long as the patent application is pending. So, if you learn of any information that fits the above criteria at any time before a patent is granted, please send it to my attention.

If you have any questions, please call to discuss them with me before signing the *Declaration & Power of Attorney*.

Please also sign the Assignment and note that it must be witnessed.

Please return the signed documents in the postage paid Express Mail envelope that we have enclosed. Please also remember that the enclosed patent application constitutes Cabot Confidential Information and should be treated by you accordingly.

Thank you for your assistance. If you have any questions or concerns, do not hesitate to let me know.

Very truly yours,

Martha Ann Finnegan

MAF/err Enclosures

# **DECLARATION AND POWER OF ATTORNEY**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below my name:

I believe I am the original, first, and joint inventor (if only one name is listed below), or an original, first, and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

# "ELASTOMER COMPOSITES, ELASTOMER BLENDS AND METHODS,"

the specification of which [ ] is attached hereto and/or	or [X] was filed as Serial No. 10/010,	<b>764</b> on
7 December 2001 and was amended on	(if applicable); or was filed a	as PCT
International Application Number	on and was ar	nended
on (if applicable).		

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United Sates Code, §§119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed:

COUNTRY (If PCT indicate PCT)	APPLICATION NO.	DATE OF FILING (day, month, year)	PRIORITY CLAIMED
			[]Yes []No

01057 PATENT

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below:

PROVISIONAL APPLICATION NUMBER(S)	FILING DATE (day, month, year)
·	
	-

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s), or of 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

U.S. PATENT AF	PLICATIONS	ST	ATUS (Check on	e)
U.S. APPLICATION NO.	U.S. FILING DATE	PATENTED	PENDING	ABANDONED
PCT APPLICATIONS DES	SIGNATING THE U.S.			
PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NO. ASSIGNED (if any)		-

As a named inventor, I hereby appoint Martha Ann Finnegan (Reg. No. 31,453), Michelle B. Lando (Reg. No. 33,941), Tim A. Cheatham (Reg. No. 40,556), and Peter M. McDermott (Reg. No. 29,411) as my attorney with full power of substitution and revocation, to prosecute this application and transact all business in the United States Patent and Trademark Office connected therewith.

Inventor's Signature:\_

Please address all communications regarding this application to:

Martha Ann Finnegan, Esq. Cabot Corporation 157 Concord Road Billerica, MA 01821

Tel: (978) 670-6198 Fax: (978) 670-8027

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full Name of First Inventor: Residence: Post Office Address:	Ting Wang Four Shepherds Lane Billerica, Massachusetts 01862 Same as above
Citizenship:	China
Inventor's Signature:	Date:
Full Name of Second Inventor:	Meng-Jiao Wang
Full Name of Second Inventor: Residence:	Meng-Jiao Wang 45 North Hancock Street Lexington, Massachusetts 02420

Full Name of Third Inventor:	Glendon A. McConnell	
Residence:	3031 LaSalle Avenue Apt. #4 Rockford, Illinois 61114	
Post Office Address:	Same as above	
Citizenship:	United States of America	
Inventor's Signature:	Date:	
Full Name of Fourth Inventor:	Steven R. Reznek	
Residence:	781 Strawberry Hill Road	
	Concord, Massachusetts 01742	
Post Office Address:	Same as above	
Citizenship:	United States of America	
Inventor's Signature:	Date:	

## **ASSIGNMENT**

We, Ting Wang, citizen of China, residing at Four Shepherds Lane, Billerica, Massachusetts 01862, U.S.A., Meng-Jiao Wang, citizen of the United States, residing at 45 North Hancock Street, Lexington, Massachusetts 02420, U.S.A., Glendon A. McConnell, citizen of the United States, residing at 3031 LaSalle Avenue, Apt. #4, Rockford, Illinois 61114, U.S.A., and Steven R. Reznek, citizen of the United States, residing at 781 Strawberry Hill Road, Concord, Massachusetts 01742, U.S.A., and respectively, having invented the subject matter described in U.S. Patent Application entitled,

# "ELASTOMER COMPOSITES, ELASTOMER BLENDS AND METHODS,"

the specification of which was filed on 7 December 2001, in the United States Patent and Trademark Office, as Serial No. 10/010,764, for good and valuable consideration, the receipt and sufficiency whereof is hereby acknowledged, hereby sell, assign, and transfer to Cabot Corporation, a Delaware corporation, having a principal place of business at Two Seaport Lane, Suite 1300, Boston, Massachusetts 02210-2019, its successors and assigns, the entire right, title and interest in and to said application and the inventions therein set forth and described, including all priority rights ensuing from said application, and any and all Letters Patent of the United States of America and of countries foreign thereto which may be granted on or for such inventions, together with the right to apply for such Letters Patent and all rights in any applications therefor. We further sell, assign and transfer to Cabot Corporation all right, title, and interest in any and all corresponding divisional, renewal, substitute, and continuation applications, and all corresponding reexamination and reissue certificates.

We hereby represent and warrant that there are no rights or interests inconsistent with the rights and interests granted herein and that we will not execute any instrument or grant or transfer any rights or interests inconsistent with the rights and interest granted herein, and we, Ting Wang, Meng-Jiao Wang, Glendon A. McConnell and Steven R. Reznek bind ourselves and our respective heirs, executors, administrators and legal representatives, as the case may be, to execute and deliver to Cabot Corporation, its successors and assigns, any further documents or instruments and do any and all further acts that may reasonably be deemed necessary by Cabot Corporation, to enable Cabot Corporation, its successors and assigns to file applications for the invention(s) in any country where it may elect to file such applications, and that may be necessary to vest in Cabot Corporation, its successors and assigns, the title herein conveyed or intended so to be, and to enable such title to be recorded in the United States and foreign countries where such application or applications may be filed.

And we further covenant and agree, in consideration of the premises, that we and our respective heirs, executors, administrators and legal representatives, as the case may be, will at any

time, upon request, communicate to **Cabot Corporation**, its successors and assigns, any facts relating to the invention and the improvements contained therein and the history thereof, known to us or our respective heirs, executors, administrators and legal representatives, as the case may be, and that we will testify as to the same in any interference or other litigation when requested so to do by **Cabot Corporation**, its successors and assigns.

, 2002.	l, Ting Wang, have hereunto set my hand this day of
	·
Ting Wang	
· ·	<del>-</del> · · · · · · · · · · · · · · · · · · ·
COMMONWEALTH/STATE OF COUNTY OF	) )
Wang, to me known and known to me	, 2002, before me personally appeared <b>Ting</b> e to be the individual described in and who executed the nowledged to me that he executed the same for the purpose
	NOTARY PUBLIC My Commission Expires:
day of, 2002.	I, Meng-Jiao Wang, have hereunto set my hand this
Meng-Jiao Wang	

•	
COMMONWEALTH/STATE OF COUNTY OF	)
Meng-Jiao Wang, to me known and	, 2002, before me personally appeared known to me to be the individual described in and who I who duly acknowledged to me that he executed the same
	NOTARY PUBLIC
	My Commission Expires:
_	
day of, 2002.	I, Glendon A. McConnell, have hereunto set my hand this
Glendon A. McConnell	
WITNESS:	
Signature	Witness Printed Name
DATE:	
	Street Address
	City, State, Zin Code

	WHEREOF, I, Steven_, 2002.	R. Reznek,	have hereunto set my hand	this
	•			
Steven R. Reznek				
COMMONWEALTH/ST	ATE OF	)		
COUNTY OF		)		
On this day of Steven R. Reznek, to mexecuted the foregoing in for the purpose therein set	e known and known t strument, and who dul	o me to be	2002, before me personally the individual described in ged to me that he executed	and who
* - <del></del>	•			
	NO	TARY PUB	LIC	
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# ELASTOMER COMPOSITES, ELASTOMER BLENDS AND METHODS (03259.00017)

#### Field of Invention

[0001] Certain aspects of the present invention relate to elastomer composites and elastomer blends. Other aspects relate to novel methods of making elastomer composites and elastomer blends.

#### Background

Numerous products of commercial significance are formed from elastomeric compositions wherein particulate filler is dispersed in various synthetic elastomers, natural rubber or elastomer blends. Carbon black, for example, is widely used as a reinforcing agent in natural rubber and other elastomers. Certain grades of commercially available carbon black are used, which vary both in surface area per unit weight and in structure, but have been limited by conventional rubber batch processing techniques. Numerous products of commercial significance are formed from such elastomeric compositions, including, for example, vehicle tires, engine mount bushings, conveyor belts, windshield wipers and the like. While a wide range of performance characteristics can be achieved employing currently available materials and manufacturing techniques, there has been a long standing need in the industry to develop elastomeric compositions having improved properties, especially elastomer compositions that can be produced effectively and economically.

#### Summary

[0003] In accordance with a first aspect of the invention, there is provided an elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer. The particulate filler comprises at least one carbon black having structure and surface area values which satisfy the equation CDBP  $\leq$  (BET  $\div$  2.9)-X, wherein X is greater than or equal to 0. Such carbon blacks are referred to in some instances below as ultra-high surface area/low structure carbon blacks. Preferably, the elastomer is natural rubber. Additional ingredients, e.g., any of numerous additives and other fillers known for use in elastomer composites, may be included in the elastomer composites, such as to achieve desired performance properties, processing characteristics etc.

[0004] In accordance with another aspect of the invention, there is provided an elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer having a tear strength, as measured for example, by test method ASTM-D624 using Die C of greater than about 160 N/mm, more preferably greater than about 165 N/mm, and most preferably greater than about 170 N/mm.

[0005] In accordance with another aspect of the invention, there is provided an elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer having a Shore A hardness, as measured for example in accordance with test method ASTM-D1415, of greater than about 65, a tensile strength, as measured for example in accordance with test method ASTM-D412, of greater than about 30 megapascals, and an elongation at break, as measured for example in accordance with test method ASTM-D412 of greater than about 600 %.

[0006] In accordance with another aspect of the invention, there is provided an elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer, wherein the particulate-filler comprises at least one carbon black in an amount such that the elastomer has tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm.

[0007] In accordance with another aspect of the invention, there is provided an elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer, wherein the particulate filler comprises at least one carbon black in an amount such that the elastomer has Shore A hardness greater than about 65, as measured in accordance with test method ASTM-D1415, tensile strength greater than about 30 megapascals, as measured in accordance with test method ASTM-D412, and elongation at break greater than about 600 %, as measured in accordance with test method ASTM-D412.

[0008] In accordance with another aspect of the invention, there is provided an elastomer composite comprising particulate filler dispersed in elastomer by methods referred to here as continuous wet mixing and coagulation wherein the carbon black has structure and surface area values satisfying the equation CDBP  $\leq$  (BET ÷ 2.9)-X, wherein X is greater than or equal to 0. Certain preferred embodiments of such elastomer composites have tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm. Certain other preferred embodiments of such elastomer composites have Shore A hardness, as measured in accordance with test method ASTM-D415, greater than about 65, tensile strength, as measured in accordance with test method ASTM-D412, greater than about 30 megapascals, and elongation at break, as measured in accordance with test method ASTM-D412, greater than about 600 %.

[0009] In accordance with another aspect of the invention, there is provided elastomer blends comprising at least one elastomer composite disclosed above blended with an elastomer material. The elastomer material comprises at least one elastomer, which may be the same as, or different from, the elastomer in the elastomer composite. Optionally, the elastomer material comprises filler, e.g., carbon black, additives or other fillers dispersed in the elastomer.

[0010] In accordance with yet another aspect of the invention, there is provided a method of preparing the elastomer composites and elastomer blends disclosed above. This method comprises compounding or mixing the elastomer and the particulate filler, including the carbon black, and optionally other ingredients. In certain preferred embodiments such elastomer composites are produced by methods comprising continuous wet mixing and coagulation. Similarly, in certain preferred embodiments such elastomer blends are produced by methods comprising continuous wet mixing and coagulation followed by further compounding or mixing with additional elastomer material comprising at least one elastomer, which may the same as, or different from, the elastomer in the elastomer composite. Optionally, the elastomer material comprises filler, e.g., carbon black, additives or other fillers dispersed in the elastomer.

[0011] These and other aspects and advantages will be further understood in view of the following detailed discussion of certain preferred embodiments.

#### **Detailed Description of Certain Preferred Embodiments**

Preferred methods and apparatus for producing elastomer composites disclosed here are described in commonly assigned United States Patent Nos. 6,075,084, 6,048,923, and 6,040,364, the entire disclosure of each of which is hereby incorporated herein by reference for all purposes. Such methods are referred to here in some instances as continuous wet mixing and coagulation. A preferred method for producing the elastomer composites comprises feeding a continuous flow of first fluid comprising elastomer latex to a mixing zone of a coagulum reactor defining an elongate coagulum zone extending from the mixing zone to a discharge end, and feeding a continuous flow of second fluid comprising particulate filler under pressure to the mixing zone of the coagulum reactor to form a mixture with the elastomer latex. The mixture passes as a continuous flow to the discharge end of the coagulum reactor, and the particulate filler is effective to coagulate the elastomer latex. More specifically, the second fluid is fed again at the first fluid within the mixing zone sufficiently energetically to substantially completely coagulate the elastomer latex with the particulate filler prior to the discharge end of the coagulum reactor. A substantially continuous flow of elastomer composite is discharged from the discharge end. As

noted above, these processes are referred to below in some instances as continuous wet mixing and coagulation.

In certain preferred embodiments, particulate filler slurry is fed to the mixing [0013] zone preferably as a continuous, high velocity jet of injected fluid, while the latex fluid typically is fed at relatively lower velocity. The high velocity, flow rate and particulate concentration of the filler slurry are sufficient to cause mixture and high shear of the latex fluid, flow turbulence of the mixture within at least an upstream portion of the coagulum zone, and substantially complete coagulation of the elastomer latex prior to the discharge end. Substantially complete coagulation can thus be achieved, in accordance with preferred embodiments, without the need of employing an acid or salt coagulation agent. Preferred continuous flow methods for producing the elastomer composites comprise continuous and simultaneous feeding of latex fluid and filler slurry to the mixing zone of the coagulum reactor, establishing a continuous, semi-confined flow of a mixture of the latex and filler slurry in the coagulum zone. Elastomer composite crumb in the form of "worms" or globules are discharged from the discharge end of the coagulum reactor as a substantially constant flow concurrently with the on-going feeding of the latex and carbon black slurry streams into the mixing zone of the coagulum reactor. Feed rates of the latex fluid and carbon black slurry to the mixing zone of the coagulum reactor can be precisely metered to achieve high yield rates, with little free latex and little undispersed carbon black in the product crumb at the discharge end of the coagulum reactor. High feed velocity of the carbon black slurry into the mixing zone of the coagulum reactor and velocity differential relative the latex fluid feed are believed to be significant in achieving sufficiently energetic shear of the latex by the impact of the particulate filler fluid jet for thorough mixing and dispersion of the particulate into the latex fluid and coagulation of the latex. Prior techniques involving premixing of latex and particulate filler, such as in the above-mentioned Heller et al patent and Hagopian et al patent, do not recognize the possibility of achieving coagulation without exposing the latex/particulate mixture to the usual coagulant solution with its attendant cost and waste disposal disadvantages. Modified and alternative suitable methods for producing novel elastomen composites disclosed here will be apparent to those skilled in the art, given the benefit of this disclosure.

[0014] Numerous carbon blacks are suitable for use in the elastomer composites disclosed here, including commercially available carbon blacks and fillers comprising carbon black. In addition to the carbon blacks specifically disclosed here, additional carbon blacks will be apparent to those skilled in the art, given the benefit of this disclosure.

[0015] In accordance with one aspect of this invention, there is provided an elastomer composite comprising carbon blacks having surface area and structure values satisfying equation

wherein X is greater than or equal to 0. Preferably X is 0, and, when X is 0, Equation (1) may also be represented herein as "CDBP  $\leq$  (BET  $\div$  2.9)."

In Equation (1), the structure value CDBP is the dibutylphthalate adsorption number after the sample has been crushed and is measured in accordance with the test procedure described in ASTM D-3493. The surface area value BET is nitrogen adsorption surface area and is measured in accordance with the test procedure described in ASTM D-4820. An example of a carbon black which satisfies this equation is BP1100, which has a CDBP value of about 43 to 45 mL/100g and a BET value of about 260 to 264 m²/g. Other preferred carbon blacks meeting Equation (1) include, for example, BP 1180, BP880, and CSX439, It has now been found that certain preferred embodiments of elastomer composites disclosed here, comprising such ultrahigh surface area/low structure carbon blacks, have advantageous performance properties and processing characteristics.

[0017] In accordance with certain preferred embodiments, elastomer composites are disclosed here comprising carbon blacks having surface area and structure values satisfying Equation (2):

$$CDBP \le (BET \div 2.9) - X \tag{2}$$

wherein X is preferably about 5. More preferred are carbon blacks having surface area and structure values satisfying Equation (2) wherein X is about 10. It will be recognized, that the carbon blacks which satisfy Equation (2) are a subset of the carbon blacks which satisfy Equation (1). Additional suitable ultra-high surface area/low structure will be apparent to those skilled in the art, given the benefit of this disclosure.

Preferably, there is at least about 60 phr of ultra-high surface area/low structure carbon black in the elastomer composite. More preferably, there is at least about 65 phr of such ultra-high surface area/low structure carbon black in the elastomer composite, e.g., at least about 70 phr of such ultra-high surface area/low structure carbon black. Particularly preferred are such elastomer composites prepared by continuous wet mixing and coagulation and elastomer blends prepared by continuous wet mixing and coagulation and follow-on dry mixing with additional elastomer and/or other fillers, additives, etc. The advantageous elastomer composite properties disclosed here, such as high tensile strength, e.g., tensile strength over 160 N/mm, and good hardness, tensile strength and elongation, for example, are not found using traditional reinforcing carbon blacks.

[0019] The tear strength of the natural rubber elastomer composites comprising ultrahigh surface area/low structure carbon black, e.g., carbon black BP1100, prepared by continuous wet mixing and coagulation exceeds the tear strength, as measured by test method ASTM-D624 using Die C, of natural rubber elastomer composites of the same formulation but produced by dry mixing. At least certain preferred embodiments of continuous wet mixing and coagulation elastomer composite comprising BP 1100 have now been found to achieve a value of 160 N/mm at less than about 65 phr filler and to exceed 160 N/mm at higher loading levels. The carbon black BP 1100 is commercially available from Cabot Corporation and, as noted above, has surface area and structure values meeting the equation CDBP ≤ (BET ÷ 2.9). More specifically, as described above, BP 1100 has a BET surface area value of about 260 to 264 m²/g and a CDBP structure value of 43 to 45 mL/100g. Also, it has now been found that the tear strength of such a BP 1100 continuous wet mixing and coagulation elastomer composite significantly exceeds the tear strengths of comparably formulated elastomer composites produced by dry-mixing with other commercially available carbon blacks not meeting the equation CDBP ≤ (BET ÷ 2.9).

[0020] Comparative tear strength data also shows that the tear strength of elastomer composites comprising ultra-high surface area/low structure carbon black BP 1100, and produced by continuous wet mixing and coagulation, exceeds the tear strength of comparable elastomer composite comprising carbon black V7H, a non-ultra-high surface area/low structure carbon black, and produced by dry-mixing. Comparative tear strength data also shows that the tear strength of elastomer composites comprising BP 1100 and produced by continuous wet mixing and coagulation exceeds, at those tested loading levels between 50 and 110 phr, the tear strength of comparable elastomer composites comprising BP 1100 but produced by dry-mixing.

At least certain elastomer composites in accordance with this disclosure are suitable to be blended with additional elastomer, filler, other additives, etc. That is, at least certain of the elastomer composites disclosed here can be blended by subsequent dry-mixing with additional elastomer and/or filler or other additives, etc., including additional elastomer composites of the present invention Elastomer materials blended with elastomer composites disclosed here optionally comprise the same or different elastomer, and optionally may have carbon black and/or other filler or other additives dispersed therein.

[0022] Elastomer composites of the invention, particularly those prepared by continuous wet mixing and coagulation, can be incorporated into elastomer blends using a variety of techniques known in the art, including by subsequent dry mixing with additional elastomer material, e.g. a second elastomer or an elastomer composite already comprising fillers and/or additives, etc. The dry mixing can be carried out with any suitable apparatus and techniques,

such as commercially available apparatus and techniques. In one embodiment, aBanbury mixer or the like is used. Other ingredients also may be added along with the additional elastomer during dry mixing, including, for example, extender oil, antioxidant, cure activators, additional particulate filler, curatives (for example, zinc oxide and stearic acid), etc. In those embodiments wherein additional filler is added during follow-on dry mixing, such additional filler can be the same as or different from filler(s) in the elastomer composite. The elastomer blends may optionally undergo further processing steps known to those skilled in the art.

[0023] Preferred methods of producing elastomer composite blend, comprising first preparing elastomer composite by continuous wet mixing and coagulation as described above, followed by dry mixing the elastomer composite with additional elastomer material to form elastomer blend, are described in U.S. Patent No. 6,075,084, the entire disclosure of which is hereby incorporated herein by reference for all purposes. This process may be referred to in some instances as continuous wet mixing and coagulation with follow-on dry mixing. As stated above, at least certain preferred embodiments of the elastomer composites disclosed here are produced by continuous wet mixing and coagulation, and at least certain preferred embodiments of elastomer blends can be produced by continuous wet mixing and coagulation with follow-on dry mixing. In accordance with certain preferred embodiments, elastomer composites are produced by such continuous wet mixing and coagulation methods and apparatus, in a continuous flow process without the need for using traditional coagulating agents, such as acids or salts.

Advantageous flexibility is achieved by the method disclosed here for making [0024] elastomer blends, comprising continuous wet mixing and coagulation with follow-on dry mixing. In particular, flexibility is provided as to the choice of elastomer(s) employed in continuous wet mixing and coagulation and in the choice of elastomer(s) used in the subsequent dry mixing step. The same elastomer or mixture of elastomers can be used in the wet and dry mixing steps or, alternatively, different elastomers can be used in any suitable relative weight proportion. Further flexibility is provided in that additional filler and other additives and the like may optionally be added during either wet mixing or dry mixing. It should be understood that the dry mixing can be a multi-stage compounding process. Such additional materials can be the same as or different from those used in the continuous wet mixing and coagulation. Without wishing to be bound by theory, it presently is understood that, in at least certain preferred embodiments, a multi-phase elastomer composite blend is produced by continuous wet mixing and coagulation with follow-on dry mixing. That is, although difficult to identify or observe using techniques currently in general use in the elastomer industry, the elastomer blend is understood to comprise at lease one elastomer phase produced by continuous wet mixing and coagulation and another elastomer phase added or produced by follow-on dry mixing. The degree of mixing or blending of the two phases and the degree to which boundary layers between the two phases are more or less distinct will depend on numerous factors, including, for example, the mutual affinity of the elastomers, the level of filler loading, the choice of filler(s) and whether additional filler is added during dry mixing, the relative weight proportion of the continuous wet mixing and coagulation elastomer and the dry mixing elastomer, etc.

Numerous elastomers suitable for use in the elastomer composites disclosed here are commercially available or are otherwise known and prepared according to known techniques. Suitable elastomers include, but are not limited to, natural rubber, which is preferred, and other rubbers and polymers (e.g., homopolymers, copolymers, terpolymers, etc., all referred to here generally as polymers or copolymers unless otherwise stated or otherwise clear from context) of 1,3-butadiene, styrene, isoprene, isobutylene, 2,3-dimethyl-1,3-butadiene, acrylonitrile, ethylene, and propylene and the like. In accordance with certain preferred embodiments, the elastomer has a glass transition temperature (Tg), as measured by differential scanning calorimetry (DSC), ranging from about -120°C to about 0°C. Examples include, but are not limited to, natural rubber and its derivatives such as chlorinated rubber, styrene-butadiene rubber (SBR), polybutadiene, polyisoprene, poly(stryene-co-butadiene) and the oil extended derivatives of any of them. Blends of any of the foregoing may also be used.

In certain preferred embodiments employing continuous wet mixing and [0026] coagulation, suitable elastomers are employed as latex fluids, e.g., natural or synthetic elastomer latices and latex blends. The latex preferably is suitable for coagulation by the selected particulate filler and must be suitable for the intended purpose or application of the final rubber product. It will be within the ability of those skilled in the art to select suitable elastomer latex or a suitable blend of elastomer latices for use in continuous wet mixing and coagulation to produce elastomer composites disclosed here, given the benefit of this disclosure. Exemplary elastomers include, but are not limited to, natural rubber and latices of the other elastomers recited above. The latex may be in an aqueous carrier liquid. Alternatively, the liquid carrier may be a hydrocarbon solvent. In any event, the elastomer latex fluid must be suitable for controlled continuous feed at appropriate velocity, pressure and concentration into the mixing zone. Suitable synthetic rubber latices include, for example, copolymers of from about 10 to about 70 percent by weight of styrene and from about 90 to about 30 percent by weight of butadiene, such as copolymer of 19 parts styrene and 81 parts butadiene, a copolymer of 30 parts styrene and 70 parts butadiene, a copolymer of 43 parts styrene and 57 parts butadiene and a copolymer of 50 parts styrene and 50 parts butadiene; polymers and copolymers of conjugated dienes such as polybutadiene, polyisoprene, polychloroprene, and the like, and copolymers of such conjugated dienes with an ethylenic group-containing monomer copolymerizable therewith such as styrene, methyl styrene, chlorostyrene, acrylonitrile, 2-vinyl-pyridine, 5-methyl-2-vinylpyridine, 5-ethyl-2-vinylpyridine, 2-methyl-5-vinylpyridine, alkyl-substituted acrylates, vinyl ketone, methyl isopropenyl ketone, methyl vinyl either, and alphamethylene carboxylic acids and the esters and amides thereof, such as acrylic acid and dialkylacrylic acid amide. Also suitable are copolymers of ethylene and other high alpha olefins such as propylene, 1-butene and 1-pentene.

Elastomers suitable for use in elastomer blends disclosed here, that is, elastomers [0027] suitable for addition to the disclosed elastomer composites, include numerous elastomers that are commercially available or are otherwise known and prepared according to known techniques. Exemplary elastomers include those listed above for use in the elastomer composites. In certain preferred embodiments of the elastomer blends, specifically, those produced by continuous wet mixing and coagulation and follow-on dry mixing with additional elastomer, the additional elastomer during the dry mixing step can be any elastomer or mixture of elastomers suitable to the intended use or application of the finished product, including those listed above for use in continuous wet mixing and coagulation. In accordance with certain preferred embodiments, the elastomer latex employed in continuous wet mixing and coagulation is natural rubber latex and the additional elastomer employed in the dry mixing step is butadiene rubber (BR). In such preferred embodiments, the butadiene rubber preferably forms the minor phase or constituent of the elastomer composite blend, most preferably being from 10% to 50% by weight of total elastomer in the elastomer composite blend. In accordance with certain other preferred embodiments, the elastomer latex employed in continuous wet mixing and coagulation is natural rubber latex and the additional elastomer employed in the dry mixing step is styrene-buta-diene rubber (SBR). In such preferred embodiments, the SBR preferably forms the major phase or constituent of the elastomer composite blend, most preferably being from 50% to 90% by weight of total elastomer in the elastomer composite blend. In accordance with certain other preferred embodiments, the additional elastomer is natural rubber. In accordance with certain other preferred embodiments, the elastomer latex employed in continuous wet mixing and coagulation is butadiene rubber latex and the additional elastomer employed in the dry mixing step is SBR. In such preferred embodiments, the SBR preferably is from 10% to 90% by weight of the total elastomer in the elastomer composite blend. In accordance with certain other pre-ferred embodiments, the elastomer latex employed in continuous wet mixing and coagulation is butadiene rubber latex and the additional elastomer employed in the dry mixing step is matural rubber. In such preferred embodiments, the natural rubber preferably is the minor constituent or phase of the elastomer composite blend, most preferably being from 10% to 50% by weight of total elastomer in the elastomer composite blend. In accordance with certain other preferred embodiments employing butadiene rubber latex in continuous wet mixing and coagulation, the additional elastomer is additional butadiene rubber. In accordance with certain other preferred embodiments, the elastomer latex employed in continuous wet mixing and coagulation is SBR and the additional elastomer is butadiene rubber. In such preferred embodiments, the butadiene rubber preferably is from 10% to 90% by weight of total elastomer in the elastomer composite blend. In accordance with certain other preferred embodiments, the elastomer latex employed in continuous wet mixing and coagulation is SBR and the additional elastomer is natural rubber. In such preferred embodiments, the natural rubber preferably is the major constituent or phase, most preferably being from 50% to 90% by weight of total elastomer in the elastomer composite blend. Certain other preferred embodiments SBR is employed in both the wet mixing and dry mixing steps, thus being essentially 100% of the elastomer in the elastomer composite blend.

... Where the elastomer latex employed in continuous wet mixing and coagulation  $[0028]^{-}$ comprises natural rubber latex, the natural rubber latex can comprise field latex or latex concentrate (produced, for example, by evaporation, centrifugation or creaming). The natural rubber latex in such embodiments preferably is suitable for coagulation by the carbon black. The latex is provided typically in an aqueous carrier liquid. Alternatively, the liquid carrier may be a hydrocarbon solvent. In any event, the natural rubber latex fluid must be suitable for controlled continuous feed at appropriate velocity, pressure and concentration into the mixing zone. well-known instability of natural rubber latex is advantageously accommodated in certain preferred embodiments employing continuous wet mixing and coagulation, wherein it is subjected to relatively low pressure and low shear throughout the system until it is entrained into a semi-confined turbulent flow upon encountering a carbon black slurry feed stream jet of high velocity and kinetic energy in the mixing zone of a coagulum reactor. In certain preferred embodiments, for example, the natural rubber is fed to the mixing zone at a pressure of about 5 psig, at a feed velocity in the range of about 3-12 ft. per second, more preferably about 4-6 ft. per second. Selection of a suitable latex or blend of latices will be well within the ability of those skilled in the art given the benefit of the present disclosure and the knowledge of selection criteria generally well recognized in the industry.

[0029] As disclosed above, certain preferred embodiments of the elastomer composites of the invention comprise carbon blacks capable of providing the elastomer composite advantageous tear strength properties. In accordance with certain preferred embodirments, elastomer composites disclosed here comprise general purpose rubber and particulate filler

dispersed in the elastomer, wherein the particulate filler comprises at least one carbon black effective or sufficient in selected concentrations in the elastomer to achieve tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm. Examples of a general purpose rubber include, but are not limited to, natural rubber, polyisoprene, polybutadiene, styrene-butadiene rubber, nitrile-butadiene rubber, or ethylene-propylene rubber (including EPDM). Preferably such general purpose rubber is natural rubber.

[0030] As disclosed above, certain preferred embodiments of the elastomer composites disclosed here comprise carbon blacks capable of providing the elastomer composite advantageous hardness, tensile strength and elongation at break. In accordance with certain preferred embodiments, elastomer composites disclosed here comprise elastomer and particulate filler dispersed in the elastomer, wherein the particulate filler comprises at least one carbon black effective or sufficient in selected concentrations in the elastomer to achieve:

Shore A hardness, measured in accordance with test method ASTM-D1415, greater than about 65;-

tensile strength, measured in accordance with test method ASTM-D412, greater than 30 megapascals; and

elongation at break, measured in accordance with test method ASTM-D412, of at least about 600%.

[0031] In certain such preferred embodiments, the elastomer composite comprises elastomer and particulate filler dispersed in the elastomer, and the elastomer composite has Shore A hardness greater than about 65, tensile strength greater than about 30 megapascals, and elongation at break greater than about 600 %. Preferred embodiments comprise at least one ultrahigh surface area/low structure carbon black, more preferably at least one ultra-high surface area/low structure carbon black.

Optionally, the filler employed in the elastomer composite comprises one or more materials additional to the aforesaid carbon black. In embodiments prepared by continuous wet mixing and coagulation, and for embodiments of the elastomer blends disclosed here prepared by continuous wet mixing and coagulation and follow-on dry mixing, the carbon black filler of the elastomer composite can include also other material that can be slurried and fed to the mixing zone in accordance with the principles disclosed here. Suitable additional materials include, for example, conductive fillers, reinforcing fillers, fillers comprising short fibers (typically having an L/D aspect ratio less than 40), flakes, etc. Thus, exemplary particulate fillers which can be employed in elastomer composites disclosed here include, for example, other carbon blacks, fumed silica, precipitated silica, coated carbon blacks such as silica-coated c arbon blacks, modified carbon blacks such as those having attached organic groups, and treated c arbon

blacks including metal-treated carbon blacks (for example silicon-treated carbon blacks), either alone or in combination with each other. Suitable modified carbon blacks include those disclosed in U.S. Patent Nos. 5,851,280, 5,672,198, 6,042,643, 5,900,029, and 5,559,169 and U.S. Patent Application Serial No. 09/257,237, the entire disclosures of which are hereby incorporated by reference for all purposes. Suitable treated carbon blacks and coated carbon blacks are known and include those disclosed in U.S. Patent Nos. 5,916,934, 5,830,930, 6,028,137, 6,008,272, 5,919,841, 6,017,980, 5,904,762, 6,057,387, and 6,211,279 and U.S. Patent Application Serial Nos. 09/392,803 and 09/813,439, the entire disclosures of which are hereby incorporated by reference herein for all purposes. For example, in such silicon-treated carbon blacks, a silicon containing species, such as an oxide or carbide of silicon, is distributed through at least a portion of the carbon black aggregate as an intrinsic part of the carbon black. Also for example, in such silicon-coated carbon blacks, a silicon containing species, such as silica, is disposed on at least a portion of the surface of the carbon black aggregates. Additional materials and additives suitable to be employed with-the aforesaid carbon blacks in the elastomer composites disclosed here will be apparent to those skilled in the art, given the benefit of this disclosure.

[0033] The rubber compositions of the present invention may optionally contain various additives along with the elastomer and filler, such as curing agents, coupling agents, and optionally, various processing aids, oil extenders and antidegradents. Examples of additives include, but are not limited to, antiozonants, antioxidants, plasticizers, resins, flame retardants, and lubricants. Combinations of additives can also be used. In that regard, it should be understood that the elastomer composites of the invention include vulcanized compositions (VR), thermoplastic vulcanizates (TPV), thermoplastic elastomers (TPE) and thermoplastic polyolefins (TPO). TPV, TPE, and TPO materials are further classified by their ability to be extruded and molded several times without substantial loss of performance characteristics. Thus, in making or further processing the elastomer composite blends, one or more curing agents such as, for example, sulfur, sulfur donors, activators, accelerators, peroxides, and other systems used to effect vulcanization of the elastomer composition may be used.

The elastomer composite produced by continuous wet mixing and coagulation may optionally undergo further processing. For example, the elastomer composite may be further processed in a mixing and compounding apparatus, such as a continuous compounder. Suitable continuous compounders are described in PCT Publication No. WO 00/62990, the entire disclosure of which is hereby incorporated herein by reference for all purposes.

[0035] A significant advantage has now been recognized in preparing elastomer composites by continuous wet mixing and coagulation. Specifically, excellent abrasion resi stance

is achieved, even in elastomer composites comprising oil to reduce hardness. Typically, hardness of an elastomer composite increases as the amount of filler in the composite is increased. Often, an intended use of an elastomer composite calls for high carbon black loading. However, the intended use of the elastomer composite may also call for lower hardness. It is known to add oil to reduce hardness of an elastomer composite, but if the amount of oil in an elastomer is increased to avoid undesirable hardness, the abrasion resistance of the elastomer composite generally is reduced. Elastomer composites prepared by continuous wet mixing and coagulation, including at least certain preferred embodiments of the elastomer composites disclosed here, achieve high abrasion resistance, notwithstanding high filler content and correspondingly high amounts of oil to control hardness. That is, abrasion resistance is found to be higher than in corresponding dry mixed elastomer composites. For example, novel elastomer composites made by continuous wet mixing and coagulation with loading levels of Vulcan 7H greater than 50 phr have higher abrasion resistance than corresponding dry mixed elastomer composite of the same formulation. It should be understood that the absolute value of abrasion resistance will depend on choice of filler, elastomer and oil, as well as filler and oil loading levels, etc. For comparable formulations, however, elastomer composites produced by continuous wet mixing and coagulation, such as certain preferred embodiments of the elastomer composites disclosed here, have advantageously higher abrasion resistance than corresponding elastomer composites of the same formulation prepared using dry mixing techniques in accordance with the best commercial practices.

Elastomer composites were prepared using continuous wet mixing and [0036] coagulation and tested for abrasion resistance. Abrasion resistance was found generally to increases to a maximum and then decreases with increasing filler loading. It is also seen that the abrasion resistance of continuous wet mixing and coagulation elastomer composites is generally greater than that of corresponding dry-mixed elastomer composite, especially at the higher carbon black loading levels. For example, the abrasion resistance of a natural rubber elastomer composite prepared using continuous wet mixing and coagulation and carbon black BP110O and H65 oil was found to be greater than the abrasion resistance of a comparable elastomer composite prepared using dry mixing methods. At 50 phr filler loading, the elastomer composite pre-pared using continuous wet mixing and coagulation exhibits a 200% increase in abrasion resistance over that of the analogous elastomer composite prepared using dry mixing. The abrasion resistance of natural rubber elastomer composites over a series of filler loading levels, pre-pared by continuous wet mixing and coagulation and employing carbon black Vulcan7H and H65 oil was found to be greater than the abrasion resistance of comparable elastomer composites prepared using dry mixing methods. The abrasion resistance of natural rubber elas tomer composites over a series of filler loading levels, prepared by continuous wet mixing and coagulation and employing carbon black BP 1100, was found to be greater than the abrasion resistance of comparable elastomer composites prepared using dry mixing methods. At 80 phr filler loading, the elastomer composite prepared using the continuous wet mixing and coagulation exhibits 300% greater abrasion resistance than that of the corresponding elastomer composite prepared by dry mixing. Thus, using the continuous wet mixing and coagulation, especially employing ultra-high surface are/low structure carbon blacks, elastomer composites having high abrasion resistance can be produced.

[0037] As used here, the carbon black structure can be measured as the dibutyl phthalate adsorption (DBPA) value, expressed as cubic centimeters of DBPA per 100 grams carbon black, according to the procedure set forth in ASTM D2414. The carbon black surface area can be measured as CTAB expressed as square meters per gram of carbon black, according to the procedure set forth in ASTM D3765-85. Measurements of BET and CDBP values are as described previously above.

#### **EXAMPLES**

[0038] For each of the following examples, elastomer composite was produced comprising natural rubber from field latex, BP1100 carbon black available from Cabot Corporation, and aromatic oil. The properties of the natural rubber field latex are provided in Table 1 below:

Table 1
Natural rubber field latex properties

Total solids content, % (m/m)	32.8
	31.9
Dry rubber content, % (m/m) (50:50 Ethanol/acetic acid)	31.9
Total alkalinity, NH3, % (g/100g latex)	0.510
,,, \( \( \) \( \)	
VFA g KOH equiv. to the VFA in 100g latex solids	0.053
Acetone extract	2.11
Mooney viscosity ML (1+4)@100°C	90
·	
Mw	1565976
Mn	1170073

[0039] The full formulation of the elastomer composite is set forth in Table 2 below.

Table 2 Formulations

Ingredient	phr
Rubber	100
BP1100	50-110
Aromatic oil	0-30
ZnO	4
Stearic Acid	2
6PPD (antioxidant)	1
TBBS (accelerator)	1.2
Sulfur	1.8

# Examples 1-3

[0040] The following procedure, which is similar to that described in U.S. Patent No. 6,048,923, was used to prepare the elastomer composites of Examples 1-3.

1. Carbon black slurry preparation

Bags of carbon black were dry ground and subsequently mixed with water in a carbon black slurry tank equipped with an agitator to form a 16.8 wt% carbon black slurry. This crude slurry was then fed to a homogenizer at an operating pressure of about 3000 psig such that the slurry was introduced as a jet into the mixing zone at a flow rate of about 780 kg/hr, to produce a finely ground carbon black slurry.

#### 2. Latex delivery

The latex, which was initially charged to a tank, was pumped to the mixing zone of the coagulum reactor. The latex flow rate was adjusted in order to obtain the desired final carbon black loading levels. Latex flow rates of between 430 to 600 kg/hr gave carbon black loading levels of between 80 and 95 phr (higher latex flow rates giving lower black loading levels). No antioxidant and oil were added in the latex.

#### 3. Carbon black and latex mixing

[0043] The carbon black slurry and latex were mixed by entraining the latex into the carbon black slurry. During entrainment, the carbon black was intimately mixed into the latex and the mixture coagulated. Soft, wet spongy "worms" of coagulum exited the coagulum reactor.

#### 4. Dewatering

[0044] The wet crumb discharged from the coagulum reactor was dewatered to 10 to 25 % moisture with a dewatering extruder (The French Oil Machinery Company, 7 inch diameter). In the extruder, the wet crumb was compressed and water squeezed from the crumb and through a slotted barrel of the extruder.

#### 5. Drying and cooling-

[0045] The dewatered crumb was dropped into a continuous mixer where it was masticated and mixed with oil and antioxidant. Product exit temperature was less than 160°C and the moisture content was about 2%.

#### Tear Strength

[0046] Tear test results for the elastomer composites of Examples 1-3 are shown in Table 3 below.

Table 3
Tear strength (Die C)

Example No.	1	2	3
Carbon black, phr	80	88	95
Aromatic oil, phr	22	0	22
Tear strength, N/mm	160	177	169

#### Comparative Examples 1-3

[0047] The elastomer composites of Comparative Examples 1-3 were prepared using a BR 1600 Banbury Mixer (Farrel). The mixing procedures are shown in Table 4.

Table 4
Dry mix preparation

	أجزاز أنتها والمتقال فتناصب والمتناز	
Stage 1	Farrel BR Banbury Mixer (1600cc), 70% fill factor, 80 rpm, 45 °C	
Time (min)	Operation	
0	Add polymer	
0.5 .	Add filler	
2.5	Add oil (if any)	
4	Sweep	
5 or 8	Dump	
	Pass through open mill 3 times	
1		
Stage 2	Farrel BR Banbury Mixer (1600 cc) 65% fill factor, 60 rpm, 45 °C.	
Stage 2 Time (min)	Farrel BR Banbury Mixer (1600 cc)	
	Farrel BR Banbury Mixer (1600 cc) 65% fill factor, 60 rpm, 45 °C.	
	Farrel BR Banbury Mixer (1600 cc) 65% fill factor, 60 rpm, 45 °C.  Operation	
	Farrel BR Banbury Mixer (1600 cc) 65% fill factor, 60 rpm, 45 °C.  Operation  Add Stage 1 compound and	
Time (min)	Farrel BR Banbury Mixer (1600 cc) 65% fill factor, 60 rpm, 45 °C.  Operation  Add Stage 1 compound and curatives	
Time (min)	Farrel BR Banbury Mixer (1600 cc) 65% fill factor, 60 rpm, 45 °C.  Operation  Add Stage 1 compound and curatives  Dump	

[0048] The resulting elastomeric composites were tested for tear strength (Die C). The results are shown in Table 5.

Table 5
Tear strength (Die C)

Tear strength (Die C)						
Comparative Example No.	1 .	2	3			
Carbon black, phr Aromatic oil, phr	80 22	90 0	100 22			
Tear strength, N/mm	93	59	94			

[0049] As can be seen comparing the results shown in Table 3 with those in Table 5, elastomer composites of the present invention have considerably higher tear strength than those prepared using a conventional dry mixing method.

[0050] Although the present invention has been described above in terms of specific embodiments, it is anticipated that other uses, alterations and modifications thereof will become apparent to those skilled in the art given the benefit of this disclosure. It is intended that the following claims be read as covering such alterations and modifications as fall within the true spirit and scope of the invention.

### What is claimed is:

[0051]

1. An elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer, the particulate filler comprising at least one carbon black having structure and surface area values meeting the equation CDBP  $\leq$  (BET  $\div$  2.9).

- 2. An elastomer composite in accordance with claim 1 comprising at least 60 phr of the particulate filler.
- 3. An elastomer composite in accordance with claim 1 comprising at least 60 phr of the carbon black meeting the equation CDBP  $\leq$  (BET  $\div$  2.9).
- 4. An elastomer composite in accordance with claim 1 wherein the particulate filler further comprises at least one additional filler material having structure and surface area values not meeting the equation CDBP  $\leq$  (BET  $\div$  2.9).
- 5. An elastomer composite in accordance with claim 1 wherein the elastomer is selected from natural rubber, a homopolymer, copolymer or terpolymer of butadiene, styrene, isoprene, isobutylene, 2,3-dialkyl-1,3-butadiene where the alkyl group is C1 to C3 alkyl, acrylonitrile, ethylene or propylene.
- 6. An elastomer composite in accordance with claim 1 wherein the elastomer comprises natural rubber.
- 7. An elastomer composite in accordance with claim 1 further comprising at least one additive selected from antiozonants, antioxidants, plasticizers, processing aids, resins, flame retardants, extender oils, lubricants, and combinations thereof.
- 8. An elastomer composite in accordance with claim 1 wherein the elastomer composite has a tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm.

- 9. An elastomer composite comprising a general purpose rubber and particulate filler dispersed in the general purpose rubber, wherein the particulate filler comprises an amount of at least one carbon black effective in said general purpose rubber to achieve tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm.
- 10. An elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer, the elastomer composite having

Shore A hardness, measured in accordance with test method ASTM-D1415, greater than about 65;

tensile strength, measured in accordance with test method ASTM-D412, greater than 30 megapascals; and

elongation at break, measured in accordance with test method ASTM-D412, of at least about 600 %;

wherein the particulate filler comprises carbon black having structure and surface area values meeting the equation CDBP  $\leq$  (BET  $\div$  2.9).

- -11. An elastomer composite in accordance with claim 10 having tear strength, as measured by test method ASTM D-624, of at least 160 N/mm.
- 12. An elastomer composite comprising a general purpose rubber and particulate filler dispersed in the general purpose rubber, wherein the particulate filler comprises an amount of at least one carbon black effective in said general purpose rubber to achieve:

Shore A hardness, measured in accordance with test method ASTM-D1415, greater than about 65;

tensile strength, measured in accordance with test method ASTM-D412, greater than 30 megapascals; and

elongation at break, measured in accordance with test method ASTM-D412, of at least about 600 %.

- 13. An elastomer composite in accordance with claim 12 wherein said carbon black has structure and surface area values meeting the equation CDBP  $\leq$  (BET  $\div$  2.9).
- 14. An elastomer composite in accordance with claim 12 wherein said elastomer has tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm.

- 15. An elastomer composite in accordance with claim 1 wherein the particulate filler comprises at least one carbon black having structure and surface area values meeting the equation  $CDBP \le (BET \div 2.9) X$ , wherein X is about 5.
- 16. An elastomer composite in accordance with claim 1 wherein the particulate filler comprises at least one carbon black having structure and surface area values meeting the equation  $CDBP \le (BET \div 2.9) X$ , wherein X is about 10.
- 17. An elastomer blend comprising an elastomer composite blended with at least one elastomer material, the elastomer composite comprising a first elastomer and particulate filler dispersed in the first elastomer, the particulate filler comprising at least one carbon black having structure and surface area values meeting the equation CDBP  $\leq$  (BET  $\div$  2.9)-X.
- 18. An elastomer blend in accordance with claim 17 wherein the elastomer material comprises elastomer different from the first elastomer.
- 19. An elastomer blend in accordance with claim 17 wherein the elastomer composite, before blending, has tear strength, as measured by test method ASTM-D624 using Die C, of at least about 160 N/mm.
- 20. An elastomer blend in accordance with claim 17 wherein the elastomer composite, before blending, has

Shore A hardness, measured in accordance with test method ASTM-D1415, greater than about 65;

tensile strength, measured in accordance with test method ASTM-D412, greater than 30 megapascals; and

elongation at break, measured in accordance with test method ASTM-D412, of at least about 600%.

21. A method of producing an elastomer composite comprising an elastomer and particulate filler dispersed in the elastomer, the method comprising:

feeding a continuous flow of first fluid comprising elastomer latex to a mixing zone; and

feeding a continuous flow of second fluid comprising the particulate filler under pressure to the mixing zone to form a mixture, the mixing of the first fluid and the second fluid within the mixing zone being sufficiently energetic to substantially completely coagulate the elastomer latex with the particulate filler;

the particulate filler comprising at least one carbon black having structure and surface area values meeting the equation CDBP  $\leq$  (BET  $\div$  2.9)-X.

- 22. The method of claim 21 wherein the elastomer composite has tear strength, as measured by test method ASTM-D624 using Die C, of at least 160 N/mm.
- 23. The method of claim 21 further comprising blending the elastomer composite with an elastomer material to form an elastomer blend.
- 24. The method of claim 23 wherein blending the elastomer composite with the elastomer material comprises dry mixing the elastomer composite with the elastomer material.
- 25. The method of claim 23 wherein the elastomer material-comprises additional filler.
- 26. The method of claim 21 further comprising blending the elastomer composite with additional filler.
- 27. The method of claim 21 wherein the elastomer composite has tear strength, as measured by test method ASTM-D624 using Die C, of at least 160 N/mm.
- 28. The method of claim 21 wherein the elastomer composite has

  Shore A hardness, as measured by test method ASTM D1415, of at least 65,
  tensile strength, as measured by test method ASTM D412, of at least 30
  megapascals, and

elongation, as measured by test method ASTM D412, of at least 600 %.

29. An elastomer composite in accordance with claim 1, wherein the particulate filler comprises at least one carbon black having a CDBP value of about 43 to 45 mL/100g and a BET value of about 260 to 264 m<sup>2</sup>/g.

### ABSTRACT OF THE DISCLOSURE

Elastomer composites and elastomer blends comprising particulate fillers having selected structure and surface area values are disclosed as well as elastomer composites and elastomer blends having improved properties. Methods of making and using elastomer composites and elastomer blends are also disclosed.

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# **EXHIBIT D**





13 June 2002

#### CABOT CONFIDENTIAL

Martha Ann Finnegan viet Intellectual Property Counsel Glendon A. McConnell 3031 LaSalle Avenue Apt. #4 Rockford, Illinois 61114

Re: U.S Patent Application Serial No. 10/010,764

Filed: 7 December 2001

Title: "Elastomer Composites, Elastomer Blends and Methods"

Our Reference No.: 01057

### Reply requested as soon as possible

#### Dear Glen:

This is further to my letter of 30 May 2002, forwarding a copy of the above-identified patent application and an original *Declaration and Power of Attorney* and *Assignment* therefor by Express Mail.

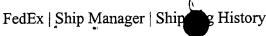
According to U.S. Postal Service records, the May 30<sup>th</sup> Express Mail package was successfully delivered to the address above on 31 May 2002. However, to date I have received no response.

Please call me at (978)-670-6198 at your earliest convenience to let me know if you have any questions related to these documents. Otherwise, please sign and return the documents to my attention in the previously enclosed postage paid Express Mail envelope as soon as possible.

Thank you for your assistance.

Very truly yours,

Martha Ann Finnegan



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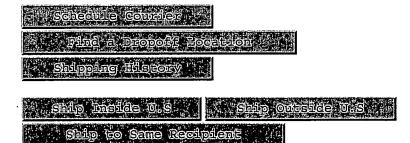
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# **EXHIBIT E**

# Martha Finnegan

## 06/21/2002 11:46 AM

To: glenmcc30@hotmail.com

cc: Louis Grasso/Billerica/Cabot@Cabot

Subject: U.S Patent Application Serial No. 10/010,764

Filed: 7 December 2001

Title: "Elastomer Composites, Elastomer Blends And Methods"

Our Reference No.: 01057

U.S Patent Application Serial No. 10/039,774

Filed: 9 November 2001

Title: "Elastomer Composite Materials in Low Density Forms and Methods"

Our Reference No.: 01058

Dear Glen,

On 31 May 2002, we forwarded the Declarations and Powers of Attorney (with copies of the respective patent applications) and related Assignments for the above-identified patent applications to you for your review and signature. Follow-up letters were sent on 13 June 2002. Such correspondence was sent to the following address, which you had provided to Human Resources:

3031 LaSalle Avenue Apt. #4 Rockford, Illinois 61114

While we have confirmed that the above correspondence was delivered to this address and signed for, to date we have not received the signed Declarations and Assignments back from you. Please confirm that you have received the four letters that we sent. Please also let us know if you have any questions. We would appreciate receiving the signed papers from you no later than next week so that we can meet the outstanding and rapidly approaching deadline. If, for any reason, you will not be able to satisfy our request, please let me know as soon as possible.

Thank you for your assistance and cooperation in this matter.

Sincerely,

Martha Finnegan Tel. No. (978) 670-6198